



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**  
OFFICE OF CRIMINAL ENFORCEMENT, FORENSICS AND TRAINING  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
P.O. BOX 25227, DENVER FEDERAL CENTER  
DENVER, COLORADO 80225

November 3, 2010

MEMORANDUM

SUBJECT: Final Criminal Technical Report [NEICRP1321R01]  
CES Environmental Services  
CID Case No.: 0606-0015  
NEIC Project No.: RP1321

FROM: (b) (6), (b) (7)(C) (b) (6), (b) (7)(C)  
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TO: (b) (6), (b) (7)(C)  
SAC, Dallas Area Office

Enclosed is one hard copy of the subject document, and a computer compact disk (CD) containing digital files of the complete report, and this transmittal memo. Please contact me at 303-462-9013, or (b) (6), (b) (7)(C) the NEIC project manager, at 303-462-9296 with any questions.

Enclosures

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United States Environmental Protection Agency  
Office of Enforcement and Compliance Assurance  
Office of Criminal Enforcement, Forensics and Training

National Enforcement Investigations Center

NEIC

**ENFORCEMENT CONFIDENTIAL**

**NEICRP1321R01**

**CRIMINAL TECHNICAL REPORT**

**CES Environmental Services**  
Port Arthur and Houston, Texas  
CID Case No.: 0606-0015  
NEIC Project No.: RP1321

November 2010

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**This Contents page shows all of the sections contained in this report and provides a clear indication of the end of this report.**



## EXECUTIVE SUMMARY

### INTRODUCTION

The U.S. Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) provided technical assistance to EPA's Criminal Investigation Division (CID), Dallas Area Office, in support of an investigation regarding the management and storage of suspected hazardous waste. NEIC personnel conducted their field activities at two CES Environmental Services (CES) facilities. Both facilities have Resource Conservation and Recovery Act (RCRA) identification (ID) numbers as large quantity generators and transporters of hazardous waste (TXR000079307 and TXD008950461).

The first facility, located at 2420 Gulfway Drive, Port Arthur, Texas, with RCRA ID No. TXR000079307, has the North American Industry Classification (NAICS) codes of 325188 (All Other Basic Inorganic Manufacturing) and 324191 (Petroleum Lubricating Oil and Grease Manufacturing)<sup>1</sup>. The second facility, located at 4904 Griggs Road, Houston, Texas, with RCRA ID No. TXD008950461, has the following NAICS codes<sup>2</sup>:

- 236220 - Commercial and Institutional Building Construction
- 488490 - Other Support Activities for Road Construction
- 562910 - Remediation Services
- 562112 - Hazardous Waste Collection
- 562219 - Other Hazardous Waste Treatment and Disposal
- 562111 - Solid Waste Collection
- 488999 - All Other Support Activities for Transport

NEIC inspector (b) (6), (b) (7)(C) obtained the identification numbers and NAICS codes from the EPA Envirofacts database.

The objective of the NEIC field assistance was to collect evidentiary samples of materials to assist in determining whether the materials exhibit RCRA hazardous waste characteristics and to document/inventory containers at the facilities. NEIC personnel—(b) (6), (b) (7)(C)—(b) (6), (b) (7)(C)—collected samples that were analyzed at the

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<sup>1</sup> The EPA Envirofacts Facility Detail Report available at [http://iaspub.epa.gov/enviro/fii\\_query\\_dtl\\_disp\\_program\\_facility](http://iaspub.epa.gov/enviro/fii_query_dtl_disp_program_facility). The report last accessed October 21, 2010.

<sup>2</sup> The EPA Envirofacts Facility Detail Report available at [http://iaspub.epa.gov/enviro/fii\\_query\\_dtl\\_disp\\_program\\_facility](http://iaspub.epa.gov/enviro/fii_query_dtl_disp_program_facility). The report last accessed October 21, 2010.

NEIC laboratory in Denver, Colorado, and at the EPA Region 6 laboratory in Houston, Texas. Stewart Simpson of NEIC conducted a partial inventory at the CES Houston facility.

The samples sent to the NEIC laboratory were analyzed for properties that identify solid waste as having the RCRA hazardous waste characteristics of corrosivity and reactivity. Analyses included measurement of pH, sulfide, water, carbon, and sulfur. The EPA Region 6 laboratory analyzed samples for properties that identify solid waste as having the RCRA hazardous waste characteristics of ignitability and toxicity; in addition, volatile and semi volatile organic constituents were determined. The EPA Region 6 laboratory results have been provided directly to EPA Region 6 Regional Criminal Enforcement Officer (RCEO) (b) (6), (b) (7) and are not included as part of this NEIC report. All field sampling and measurements performed by NEIC personnel were conducted in accordance with the NEIC quality system.

## SUMMARY OF ACTIVITIES AND FINDINGS

During the field activities performed August 4 through 6, 2009, at the CES Port Arthur facility, NEIC personnel conducted the following activities:

- Documented site conditions in and around the facility
- Accompanied (b) (6), (b) (7) in evaluating waste processing units
- Collected samples from eight stations, including one quality control replicate
- Inventoried containers

During the field activities performed August 6 through 8, 2009, at the CES Houston facility, NEIC personnel performed the following activities:

- Collected samples from 19 stations, including one quality control replicate
- Inventoried containers

The NEIC laboratory analyzed many, but not all, of the samples collected by NEIC field personnel. Some of the samples collected were sent directly to the EPA Region 6 laboratory in Houston. A number of the samples sent to and analyzed by the Houston laboratory were later sent to the NEIC laboratory for additional testing. This report contains results only from the NEIC laboratory analyses.

### CES Port Arthur Facility

- NEIC analyzed liquid samples from seven stations collected at the Port Arthur facility. These stations are: S1A (frac tank 1007), S1B (tank NV2), S2A (frac tank 1003), S2B (tank NV1), S3A (tanker 261), S4A (tanker 419), and S5A (tanker 262). In addition, one

air sample (S2A) was collected at the Port Arthur facility. The analytical results are presented in tables in the “Laboratory Activities” section of this report.

- Four samples were collected for each of stations S3A, S4A, and S5A. Each of the 12 samples had a pH of greater than 13.<sup>3</sup>
- Twelve samples from station S1B were analyzed at the NEIC laboratory. Seven of the 12 samples had a pH of greater than 12.5. The measured pH values ranged from 11.52 to greater than 13.
- All four samples from station S2A had high levels of sulfide. The measured concentrations ranged from 952 to 5,470 milligrams per kilogram (mg/kg). One sample from station S1A had a sulfide concentration of 101 mg/kg, and the laboratory composite had a concentration of 60 mg/kg. The other three samples from this station contained <1 mg/kg sulfide. One sample from station S1B contained 46.2 mg/L sulfide, and another contained 14.9 mg/kg. The other 10 samples contained <1 mg/kg sulfide.<sup>4</sup>
- One air sample was collected at station S2A. No hydrogen sulfide was detected, which is consistent with the high pH of the liquid. Dimethyl disulfide was detected and determined to have a concentration of 65 parts per million (ppm) by volume.

#### CES Houston Facility

- NEIC analyzed liquid samples from seven stations collected at the Houston facility. These stations are: S3B (tank 408), S4B (tank 407), S6A (tanker 413), S7A (tanker 235), S8A (tanker 265), S9A (frac tank 1004), and S10A (frac tank 1002).
- In addition to the samples collected by the NEIC field team, G. Tidmore collected samples that were analyzed by the NEIC laboratory. These are labeled S1C-01 through S1C-08.
- Two samples from each of the eight S1C stations were analyzed. All the samples had a pH of less than 1.<sup>3</sup> All samples contained hydrochloric acid, and S1C-07 also contained sulfuric acid.
- Station S10A samples contained sulfides in the range of 2.51 to 20.8 mg/L, and the laboratory composite contained 4.51 mg/kg.<sup>4</sup>

The results for some stations, including S1A, S1B, S2A, and S10A, indicate differences among the contents of the sample containers from the same stations. This is attributed to stratification within the sampled vessel. The field sample was the entire liquid column retrieved

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<sup>3</sup> According to 40 Code of Federal Regulations (CFR) 261.22 (a) (1), a solid waste that is an aqueous liquid with pH values less than or equal to 2.0, or greater than or equal to 12.5, exhibits the RCRA hazardous waste characteristic of corrosivity (EPA Hazardous Waste No. D002).

<sup>4</sup> According to 40 CFR 261.23 (a) (5), a solid waste exhibits the RCRA hazardous characteristic of reactivity if a representative sample of the waste is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment. A solid waste that exhibits the characteristic of reactivity has the EPA Hazardous Waste No. D003.



with the sampling device, the volume of which exceeded the volume of an individual sample container; thus, the field sample went into multiple sample containers.

## FIELD ACTIVITIES

### ONSITE ACTIVITIES

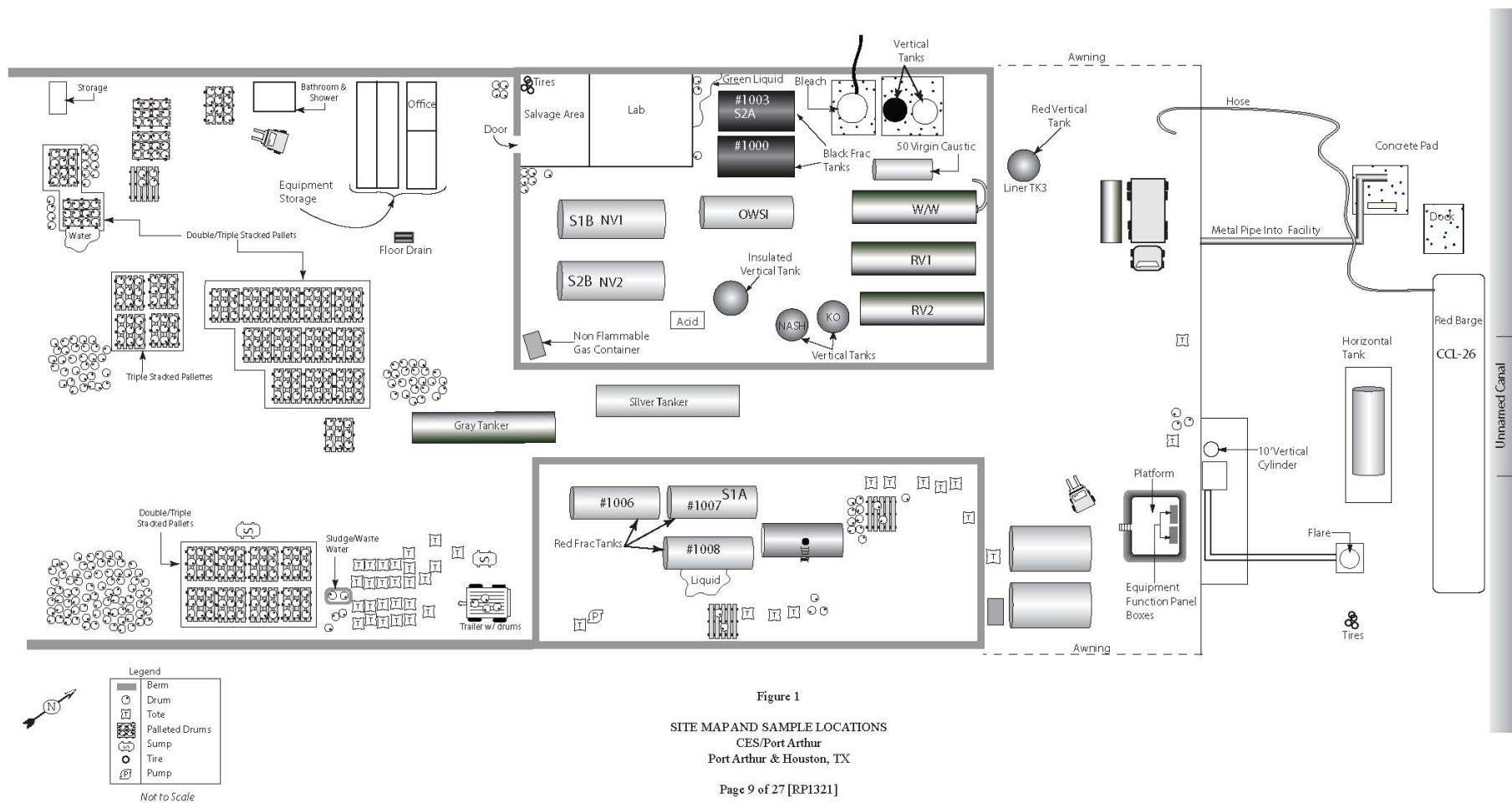
#### CES Port Arthur

During the period of August 4 through 6, 2009, the NEIC field team of (b) (6), (b) (7)(C) collected evidentiary samples at the CES Port Arthur facility. Also present were OCEFT special agents (SAs) (b) (6), (b) (7)(C); EPA Region 6 RCEO (b) (6), (b) (7)(C); EPA Region 6 On-Scene Coordinator (OSC) (b) (6), (b) (7)(C); and Superfund Technical Assessment and Response Team (START) contractor staff from Weston Solutions, Inc.

(b) (6), (b) (7)(C) conducted a limited inventory of material stored inside and around the facility [Figure 1]. (b) (6), (b) (7)(C) observed ten tanker trucks (tankers) west of the warehouse; one aboveground storage tank (AST) labeled “Dragon, 1-800-231-8198,” with an affixed placard indicating the contents were flammable; and four tanker trucks along the southern fence line. West of the warehouse and next to the loading dock area, (b) (6), (b) (7)(C) observed six small roll-off dumpsters with CES identifiers. (b) (6), (b) (7)(C) assigned a unique NEIC identifier to each of the six roll-off dumpsters inventoried. (b) (6), (b) (7)(C) observed black oily liquid flowing out of the eastern end of NEIC-designated roll-off dumpsters 1 and 3 (CES Identifiers 632 and 618, respectively). A small white bucket was under a valve on the bottom of NEIC-designated roll-off dumpster 4 (CES Identifier 617). The containers identified and inventoried are listed in Appendix A. (b) (6), (b) (7)(C) photographed all the site activities except for the sampling.

(b) (6), (b) (7)(C) observed a red barge (labeled “CCL-26”) northeast of the facility. Adjacent to the facility’s unnamed canal was a series of valves attached to a metal standpipe. Northeast of the facility was a placarded grey horizontal tank, a flare that was not operational, and equipment that appeared to be part of the process operations. (b) (6), (b) (7)(C) observed tanks of various sizes, drums, and a laboratory inside the facility [Figure 1]. The container labeling shown in Figure 1 was obtained from labels on the side of the respective units. (b) (6), (b) (7)(C) identified units 1000, 1003, 1006, 1007, and 1008 as frac tanks.

(b) (6), (b) (7)(C) accompanied (b) (6), (b) (7)(C) in evaluating the waste processing units at the site. During the site visit and visual observations, no ongoing waste processing was occurring. CES employee (b) (6), (b) (7)(C) accompanied (b) (6), (b) (7)(C) and (b) (6), (b) (7)(C) during the process discussion.



Following discussions with (b) (6), (b) (7)(C) identified the following units for screening of potential hazardous waste characteristics: tanks NV1 and NV2; frac tanks 1000, 1003, 1006, 1007, and 1008; and tankers 261, 262, and 419. The headspace of each unit was screened with a Jerome hydrogen sulfide analyzer (serial No. 1371), while pH indicator paper was used for the liquids. Two teams, consisting of personnel from HSD and NEIC, conducted the screening. The team led by K. Kegler (NEIC designated as Team A) screened tankers 261, 262, and 419 and frac tanks 1000, 1003, 1006, 1007, and 1008; the team led by (b) (6), (b) (7)(C) (NEIC designated as Team B) screened tanks NV1 and NV2 [Table 1]. Unless otherwise noted, the team led by (b) (6), (b) (7)(C) included (b) (6), (b) (7)(C) the team led by (b) (6), (b) (7)(C) included (b) (6), (b) (7)(C) (b) (6), (b) (7)(C).

**Table 1. CES PORT ARTHUR FIELD SCREENING RESULTS**  
**CES Environmental Services**  
**Port Arthur, Texas**

<b>CES Identifier</b>	<b>Field Screening</b>	<b>Tank Content Descriptions</b>
NV1	2 to 13.2 ppm hydrogen sulfide (H <sub>2</sub> S)	Dark liquid
NV2	pH >13	Dark, nonviscous liquid
1000	1.4 ppm H <sub>2</sub> S	Dark black oil
1003	7.1 ppm H <sub>2</sub> S	Dark liquid with oil layer
1006	Nothing above background	Oily dark liquid
1007	Neutral pH	Brown viscous liquid
1008	Neutral pH	Yellowish-brown liquid
261	pH > 13	Green liquid with black tint
262	2.3 ppm H <sub>2</sub> S	Greenish black liquid
419	Corrosive pH (basic)	Dark-purple to black liquid

On the basis of screening results and discussions with (b) (6), (b) (7)(C), the previously identified teams collected samples from the following units: frac tanks 1003 and 1007; tanks NV1 and NV2; and tankers 261, 262, and 419 [Table 2]. Samples collected by the team led by (b) (6), (b) (7)(C) were labeled with the station number followed by an A; while those collected by the team led by (b) (6), (b) (7)(C) were labeled with the station number followed by a B. (b) (6), (b) (7)(C) and (b) (6), (b) (7)(C) collected the samples from tankers 261, 262, and 419. Team members collected the samples using a tank composite liquid waste sampler (COLIWASA) or a “sludge judge.” Both devices are tube-like samplers that collect a full column of liquid. Aliquots from the sampling devices were placed into 8-ounce glass jars if the samples were to be analyzed at the NEIC laboratory or in 16-ounce jars if the samples were to be analyzed at the EPA Region 6 laboratory. Multiple samples containers were used to contain the entire liquid column retrieved with the sampling device.

Each team member changed gloves between the collection of each sample and used dedicated sampling equipment at each location. NEIC used the facility’s numbering system to designate the

sample locations. START personnel provided decontamination assistance to the sampling teams.

An air sample was collected from frac tank 1003 (S2A) into a stainless steel canister. A piece of copper tubing equipped with a filter was attached to the canister and lowered into the opening of frac tank 1003. The team led by (b) (6), (b) (7)(C) collected the air sample.

**Table 2. CES PORT ARTHUR FIELD SAMPLE DESCRIPTIONS**  
CES Environmental Services  
Port Arthur, Texas

NEIC Station No.	NEIC Sample Tag No.	Sample Collection Date and Time	Sample Station Location	Field Sample Description
S1A <sup>1</sup>	NE25863 NE25864 NE25865 NE25866	8/5/2009 1056 hours	Frac tank 1007	Dark-brown, viscous liquid
S2A <sup>1</sup>	NE25867 NE25868 NE25869 ME25870	8/5/2009 1618 hours	Frac tank 1003	Dark liquid with oil layer
S2A	NE25871	8/5/2009 1614 hours	Frac tank 1003	Air sample
S3A	NE25895 NE25896 NE25893 NE25894	8/6/2009 0944 hours	Tanker 261	Green liquid with black tint
S4A	NE25897 NE25898 NE25899 NE25900	8/6/2009 0954 hours	Tanker 419	Dark-purple to black liquid
S5A	NE25884 NE25885 NE25886 NE25887	8/6/2009 1007 hours	Tanker 262	Greenish-black liquid
S1B <sup>2</sup>	NE25872 NE25873 NE25874 NE25875 NE25876 NE25877 NE25878 NE25879 NE25880 NE25881 NE25882 NE25883	8/5/2009 1450 hours	Tank NV2	Dark nonviscous liquid
S2B	NE25888 NE25889 NE25890 NE25892	8/6/2009 0918 hours	Tank NV1	Hot dark liquid
<sup>1</sup> Samples collected using a sludge judge. <sup>2</sup> Samples represent quality control replicates.				

## CES Houston

On August 4 and 5, 2009, NEIC inspector (b) (6), (b) (7)(C) provided technical support and conducted a site evaluation at the CES Houston facility. (b) (6), (b) (7)(C) was accompanied by CES supervisor (b) (6), (b) (7)(C) during the evaluation of the CES Houston facility. On August 4, 2009, at the CES Houston facility, a roll-off dumpster containing material that was being processed caught fire [Appendix B – Photographs 1, 3, and 4]. The fire was extinguished by the Houston Fire Department using “Red Alert” firefighting foam that contained 2-butoxyethanol ethoxylated alkyl sulfates based on information obtained by (b) (6), (b) (7)(C) during the firefighting activities. A sample (SS1) of the runoff associated with the fire and firefighting activities was collected on August 4, 2009, by a member of the Houston Fire Department.

The combined NEIC/HSD team, OSC, and START contractor that provided technical support to CID at the CES Port Arthur facility also provided technical support to CID at the CES Houston facility during the period of August 6 through 8, 2009. Upon arrival at the site, EPA personnel were joined by Occupational Safety and Health Administration (OSHA) representatives and by (b) (6), (b) (7)(C) with the Texas Department of Environmental Quality (TDEQ). The NEIC/HSD team conducted a site survey with CID SAs to determine the most appropriate location for equipment staging, and identified those containers of concern based on background information. While staging the equipment, the team detected organic vapors near the area identified during the site survey as the used oil tank farm. Eight ASTs were present within this area, in addition to a hydrocarbon processing facility. HSD personnel used an infrared (IR) camera and tape measure to document the liquid levels within the eight ASTs, all of which were 20 feet 2 inches tall [Table 3]. (b) (6), (b) (7)(C), accompanied by SAs (b) (6), (b) (7)(C) and (b) (6), (b) (7)(C), screened the headspace within the used oil tanks with a toxic vapor analyzer (TVA) (serial No. 823340), as shown in Table 3. (b) (6), (b) (7)(C) used the facility designation to identify the tanks.

**Table 3. CES HOUSTON USED OIL TANKS FIELD SCREENING**  
CES Environmental Services  
Houston, Texas

Tank No.	Depth to Layers (from top of lid)	Field Screening Results
1	1 <sup>st</sup> layer - 6' 2 <sup>nd</sup> layer - 17'8"	14%-lower explosive limit (LEL) 11 ppm CO 115 ppm VOC 0 ppm H <sub>2</sub> S TVA/flame ionization detector (TVA/FID) 30,000 ppm



**Table 3. CES HOUSTON USED OIL TANKS FIELD SCREENING**  
**CES Environmental Services**  
**Houston, Texas**

Tank No.	Depth to Layers (from top of lid)	Field Screening Results
2	1 <sup>st</sup> layer - 8'8" 2 <sup>nd</sup> layer - 19'1"	8% LEL 144 ppm VOC 125 ppm CO 0 ppm H <sub>2</sub> S TVA/FID 15,400 ppm
3	1 <sup>st</sup> layer - 2' 2 <sup>nd</sup> layer - 18'5" 3 <sup>rd</sup> layer-not recorded	30% LEL VOC (Instrument Not responding) 234 ppm CO 0 ppm H <sub>2</sub> S TVA/FID 12,900 ppm
4	1 <sup>st</sup> layer - 6'7" 2 <sup>nd</sup> layer - 8'2" 3 <sup>rd</sup> layer - 18'4"	70% LEL 117 ppm CO TVA/FID 70,000 ppm
5	Layer - 17'7"	0 % LEL 100 ppm VOC 32 ppm CO 0 H <sub>2</sub> S TVA/FID 204 ppm
6	1 <sup>st</sup> layer - 15'2" 2 <sup>nd</sup> layer - 18'3"	100% LEL TVA/FID 96,000
7	1 <sup>st</sup> layer - 1'1" 2 <sup>nd</sup> layer - 3' 3 <sup>rd</sup> layer - 18'6"	0 % LEL 48 ppm VOC 5 ppm CO 0 ppm H <sub>2</sub> S TVA/FID 1,800 ppm
8	Layer - 18'1"	3% LEL 41 ppm VOC 2 ppm CO 0 ppm H <sub>2</sub> S TVA/FID 1,000 ppm

The field teams also used a TVA (serial No. 823340) and two MultiRAE Plus 4-Gas meters (serial Nos. 095-522090 and 095-522068), to screen the headspace within tanks identified during the initial site survey as requiring further investigation; the results of the screening are presented in **Tables 3 and 4**. The team led by (b) (6), (b) (7)(C) screened tanks 407 and 408; the team led by (b) (6), (b) (7)(C) screened tankers 235 and 265; tank 413; and frac tanks 1001, 1002, and 1004 [**Table 4**].

**Table 4. CES HOUSTON TANK SCREENING RESULTS**  
**CES Environmental Services**  
**Houston, Texas**

<b>Tank ID No.</b>	<b>Tank Fullness</b>	<b>Field Screening</b>	<b>Tank Content Descriptions</b>
235	½ full	100% LEL	Colorless liquid with yellow tint
265	Not recorded	4.4 ppm VOC 230 ppm CO	Colorless liquid
407	Not recorded	143 ppm VOC 539 and 684 ppm CO 41% LEL 25 ppm H <sub>2</sub> S	Black viscous liquid
408	Not recorded	320 ppm VOC 91 ppm CO 25% LEL 10 ppm H <sub>2</sub> S	Multi-phased brown and tan liquid
413	Full	3 ppm VOC 18.3% Oxygen	Colorless liquid with light brown tint
1001	4 feet	14 ppm VOC 0 ppm CO 0 ppm H <sub>2</sub> S	Colorless liquid with light sheen
1002	3 feet or 1/3 full	Not recorded	Black oily viscous liquid with fine silt
1004	4 feet or 1/2 full	11.5 ppm VOC 1.9 ppm CO	Black oily liquid and sludge
<sup>1</sup> Tank contents description obtained from photograph.			

On the basis of discussions with CID and EPA Region 6 personnel and screening results, the following units were identified for sampling for hazardous waste: frac tanks 1002 and 1004; tankers 235 and 265; tanks 407, 408, and 413; and used oil tanks 1, 3, 4, and 7 [Table 5]. The samples from the used oil tanks were collected using bacon bombs; a dipper cup was used to collect the sample from tank 407; a sludge judge was used to collect samples from tanks 1002 and 1004; and tank COLIWASAs were used to collect the remaining samples. For all sampling conducted, except from the used oil tanks, the teams were the same as previously described. During the sampling conducted from the used oil tanks, sample stations ending with an “A” were collected by the team of (b) (6), (b) (7)(C), and Abat, while those labeled with a “B” were collected by the team of (b) (6), (b) (7)(C), and (b) (6), (b) (7)(C). The samples collected for analysis at the NEIC laboratory were placed into 8-ounce glass jars, while those collected for analysis at the EPA Region 6 Laboratory were placed into 16-ounce glass jars. The individual sample containers did not receive the entire liquid column retrieved with the sampling device. The teams used the same procedures as previously described for the collection of the samples at the CES Port Arthur facility.

**Table 5. CES HOUSTON FIELD SAMPLE DESCRIPTIONS**  
**CES Environmental Services**  
**Houston, Texas**

<b>NEIC Station No.</b>	<b>NEIC Sample Tag No.</b>	<b>Sample Collection Date and Time</b>	<b>Sample Location/Sampling Method</b>	<b>Field Sample Description<sup>1</sup></b>
S6A	NE25902 NE25907 NE25905 NE25906	8/7/2009 0912 hours	Tanker 413/COLIWASA	Colorless liquid with light-brown tint
S7A	NE25903 NE25904 NE25908 NE25910	8/7/2009 0923 hours	Tanker 235/COLIWASA	Colorless liquid with yellow tint
S8A	NE25789 NE25909 NE25788 NE25901	8/7/2009 0933 hours	Tanker 265/COLIWASA	Colorless liquid
S9A	NE25798 NE25799 NE25800 NE25801	8/7/2009 1539 hours	Frac tank 1004/Sludge Judge	Black oily liquid and sludge
S10A <sup>2</sup>	NE25803 NE25804 NE25805 NE25806 NE25807 NE25808 NE25809 NE25810 NE25811 NE25812 NE25813 NE25814	8/7/2009 1542 hours	Frac tank 1002/Sludge Judge	Black oily viscous liquid with fine silt
S11A	NE25824 NE25825	8/8/2009 0839 hours	Used Oil Tank Farm, Tank 7, Layer 1/Bacon Bomb	Dark-black viscous liquid
S12A	NE25826	8/8/2009 0844 hours	Used Oil Tank Farm, Tank 7, Layer 2/Bacon Bomb	Dark-black sediment and viscous liquid
S13A	NE25827	8/8/2009 0851 hours	Used Oil Tank Farm, Tank 7, Layer 3/Bacon Bomb	Dark-black viscous liquid and sediment
S14A	NE25828 NE25829	8/8/2009 0901 hours	Used Oil Tank Farm, Tank 3, Layer 1/Bacon Bomb	Dark-black oil on top of light brown
S15A	NE25835 NE25836	8/8/2009 0905 hours	Used Oil Tank Farm, Tank 3, Layer 2/Bacon Bomb	Black oil on top of light brown liquid
S16A	NE25838 NE25839	8/8/2009 0909 hours	Used Oil Tank Farm, Tank 3, Layer 3/Bacon Bomb	Black oil on top of light brown liquid
S3B	NE25790 NE25791 NE25792 NE25793	8/7/2009 0857 hours	Tank 408/COLIWASA	Multi-phased brown and tan liquid <sup>1</sup>

**Table 5. CES HOUSTON FIELD SAMPLE DESCRIPTIONS**  
CES Environmental Services  
Houston, Texas

NEIC Station No.	NEIC Sample Tag No.	Sample Collection Date and Time	Sample Location/Sampling Method	Field Sample Description <sup>1</sup>
S4B	NE25794 NE25795 NE25796 NE25797	8/7/2009 0915 hours	Tank 407/Dipper Cup	Black viscous liquid <sup>1</sup>
S5B	NE25830 NE25831	8/8/2009 0837 hours	Used Oil Tank Farm, Tank 4, Layer 1/Bacon Bomb	Dark liquid
S6B	NE25832 NE25833	8/8/2009 0842 hours	Used Oil Tank Farm, Tank 4, Layer 2/Bacon Bomb	Nonviscous light liquid
S7B	NE25834 NE25840	8/8/2009 0850 hours	Used Oil Tank Farm, Tank 4, Layer 3/Bacon Bomb	Nr <sup>3</sup>
S8B	NE25841 NE25842	8/8/2009 0902 hours	Used Oil Tank Farm, Tank 1, Layer 1/Bacon Bomb	Dark liquid
S9B	NE25843 NE25844	8/8/2009 0905 hours	Used Oil Tank Farm, Tank 1, Layer 2/Bacon Bomb	Dark liquid
SS1	NE25891	8/4/2009 1735 hours	Drippings from burning roll-off dumpster	Gravel material and cloudy liquid
<sup>1</sup> Sample description obtained from photograph. <sup>2</sup> Samples represent quality control replicates. <sup>3</sup> Not recorded in field logbook.				

(b) (6), (b) (7)(C) and (b) (6), (b) (7)(C) photographed the sampling processes at both facilities [Appendix B]. The photographs were digitally stored to a Compact Flash media card. All sampling and personal protective equipment generated during the site investigation were bagged and disposed of by Superfund contractor personnel.

## EVIDENCE MANAGEMENT SUMMARY

While the team sampled and conducted field measurements, the samples were secured in locked ice chests. The samples collected at the CES Port Arthur and Houston facilities were prepared by affixing a completed sample tag to each container and placing the sample into custody bags for transportation. The samples that were analyzed at the NEIC laboratory in Denver, Colorado, were driven by (b) (6), (b) (7)(C) along with a completed chain-of-custody form [Appendix C] to the attention of Principal Analytical Chemist (b) (6), (b) (7)(C). Custody of the samples to be analyzed at the EPA Region 6 laboratory in Houston, Texas, was transferred to (b) (6), (b) (7)(C) for transportation to the EPA Region 6 laboratory [Appendix C]. (b) (6), (b) (7)(C) prepared a receipt for all the samples that was signed by (b) (6), (b) (7)(C) [Appendix D]. Custody of the samples after analysis by the EPA Region 6 laboratory was transferred to (b) (6), (b) (7)(C) on February 25, 2010, who drove them to the NEIC laboratory for additional analysis [Appendix C].

## LABORATORY ACTIVITIES

### SAMPLE DELIVERY, RECEIPT, AND TRANSFERS

The project samples were received at NEIC in two phases, as shown in **Table 6**. The phase 1 samples were received in August 2009. They were transported directly from the collection sites to NEIC. The phase 2 samples were first sent to the EPA Region 6 laboratory for some tests, and then transported by NEIC staff member (b) (6), (b) (7)(C) to NEIC. All samples were handled in accordance with the NEIC operating procedure *Evidence Management*, NEICPROC/00-059R2. Copies of the chains of custody are in **Appendix C**.

**Table 6. SAMPLE DELIVERY, RECEIPT, AND TRANSFERS**  
CES Environmental Services  
Houston and Port Arthur, Texas

Event	Date	Comment
Receipt of phase 1 samples at NEIC	August 10, 2009	(b) (6), (b) (7) of NEIC transferred custody of the phase 1 samples to (b) (6), (b) (7) at the NEIC laboratory, who placed the samples in custody in a locked cooler.
Transfer of custody of phase 2 samples	February 25, 2010	(b) (6), (b) (7) of NEIC received custody of the phase 2 samples from (b) (6), (b) (7) of EPA Region 6 laboratory.
Receipt of phase 2 samples at NEIC	March 3, 2010	(b) (6), (b) (7) transferred custody of the phase 2 samples to (b) (6), (b) (7) at the NEIC laboratory. Samples were stored in a locked ice chest in a locked cooler at the NEIC laboratory.

### BACKGROUND

The objective of the testing performed by the NEIC laboratory was to determine if any of the samples had the properties that identify solid waste as exhibiting the RCRA hazardous waste characteristics of corrosivity or reactivity. Corrosivity testing included pH measurements, while reactivity testing included sulfide determinations. A number of other tests were performed in order to more completely address the provisions of the hazardous waste regulations. **Table 7** shows the analytical procedures, the analysts, and the dates testing was performed. All analyses were conducted by NEIC personnel in accordance with the NEIC quality system.

Contents of all sample bottles received at NEIC containing samples collected at the two sites were analyzed. Each bottle had a distinguishing tag. For each station except SS1, multiple samples were analyzed. The sample from station SS1 was a sample of opportunity, and the only analysis done was the determination of the water content.

**Table 7. ANALYTICAL PROCEDURES, ANALYSTS, AND DATES**  
**CES Environmental Services**  
**Houston and Port Arthur, Texas**

Procedure(s)	Analyst(s)	Dates of Analyses
Physical description using NEIC operating procedure <i>Physical Description/Phase Separation</i> , NEICPROC/00-045R2. Physical descriptions are in the project file	Phase 1: (b) (6), (b) (7)(C)	August 2009
	Phase 2: (b) (6), (b) (7)(C)	March 2010
pH determination according to EPA SW-846 Method 9040C, <i>pH Electrometric Measurement</i> , and NEIC operating procedure <i>Potentiometric pH Determination of Aqueous Samples and Extracts</i> , NEICPROC/00-070R3	Phase 1: (b) (6), (b) (7)(C)	October and November 2009
	Phase 2: (b) (6), (b) (7)(C)	April and May 2010
Sulfide determination using Conway diffusion cells and the flow injection gas diffusion method (Milosavljevic, et al., <i>Anal. Chem.</i> 1988, vol. 60, pp. 2791 – 2796). Guidance obtained by adapting two cyanide methods for sulfide: (1) EPA Draft Method 9016, <i>Free Cyanide in Water, Soils and Solid Wastes by Microdiffusion</i> , and (2) ASTM Method D4282-02, <i>Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion</i>	Phase 1: (b) (6), (b) (7)(C) and (b) (6), (b) (7)(C)	August through October 2009
	Phase 2: (b) (6), (b) (7)(C) and (b) (6), (b) (7)(C)	March 2010
	Composites: (b) (6), (b) (7)(C)	July and August 2010
Water content determination according to EPA SW-846 Method 9000, <i>Determination of Water in Waste Materials by Karl Fischer Titration</i> , and NEIC operating procedure <i>Water Content Determination by Coulometric Karl Fisher Titration</i> . NEICPROC/00-073R2	Phase 1: (b) (6), (b) (7)(C)	August through October 2009
	Phase 2: (b) (6), (b) (7)(C)	March 2010
Anion determination for acidic samples by ion chromatography according to EPA Water Method 300.0 (Part A), <i>Determination of Inorganic Anions by Ion Chromatography</i>	(b) (6), (b) (7)(C)	April 2010
Carbon and sulfur determinations according to NEIC operating procedure <i>Elemental Analysis</i> , Appendix H, <i>Eltra CS500 Carbon and Sulfur Analyzer</i> , NEICPROC/00-062R4	(b) (6), (b) (7)(C)	Phase 1: October 2009
		Phase 2: June 2010
Analysis of one air sample according to NEIC operating procedure <i>Gas Phase Testing for Organic Contaminants</i> , NEICPROC/00-0011R4, based on EPA Method TO-15, <i>Determination of Volatile Organic Compounds in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry</i>	(b) (6), (b) (7)(C)	August 2009 and November 2009



## RESULTS FOR PH DETERMINATION

The samples were analyzed for pH using EPA SW-846 Method 9040C and NEIC procedures, as shown in Table 7. The contents of each bottle for each station were analyzed. The results of the pH determinations are summarized in **Table 8**. One sample, NE25876 from station S1B, had only enough volume for one pH determination. The other pH results are the average of multiple determinations. Samples with a high pH were diluted to check for sodium ion interferences, and samples with a low pH were diluted to check for concentrated acid interferences. Additional tests, such as titrations, were done in order to check for a variety of possible interferences with the pH determination. The results from the sample dilutions and other tests indicated that the samples were amenable to potentiometric pH measurement.

## DATA QUALITY SUMMARY FOR PH DETERMINATION

Quality control measures for the pH determination included calibration with National Institute of Standards (NIST) traceable buffers, independent calibration verification, confirmation that calibrations were valid before and after sample measurements, and replicate measurements. Instrument capability at higher pH values was checked with solutions of potassium hydroxide and sodium hydroxide prepared at NEIC and with a commercial pH 13 standard.

The replicate determinations provide an estimate of standard uncertainty. The average standard deviation for the replicate pH determinations was 0.06 standard units. The range of standard deviations was from 0.009 to 0.285 standard units.

## ANION DETERMINATION IN ACIDIC SAMPLES

Samples from eight stations with pH measurements of less than 2 were analyzed to determine the type of acid in the samples. These samples were from the stations labeled S1C-01 to S1C-08. The tag numbers are shown in Table 8, along with the acid anions that were identified. All of the samples contained chloride in amounts ranging from 15.3 to 31.6 weight percent, indicating the presence of hydrochloric acid. The two samples from station S1C-07 also contained significant amounts of sulfate, 12.7 percent and 13.1 weight percent, indicating the presence of sulfuric acid in addition to the hydrochloric acid. The chloride and sulfate were determined by ion chromatography using EPA Water Method 300.0 (Part A).

## DATA QUALITY SUMMARY FOR ANION DETERMINATION

Quality control measures for the anion determinations included blanks, known additions (spikes), independent calibration verification, continuing calibration verification, and replicate analyses.

The uncertainty of the anion determinations was estimated from the replicate determinations, the spike recoveries, and the continuing calibration standards. The standard uncertainty, expressed as a relative standard deviation, is estimated to be 3 percent.

**Table 8. ANALYTICAL RESULTS**  
**CES Environmental Services**  
**Houston and Port Arthur, Texas**

NEIC Station No.	NEIC Tag No.	pH <sup>1</sup>	Sulfide <sup>2</sup> (mg/L except as noted)	Water (percent by weight)	Carbon (percent by weight)	Sulfur (percent by weight)	Anions (percent by weight)
S1A	NE25863	5.6	0.29	53	20.8	0.172	NA
	NE25864	6.6	101 mg/kg <sup>3</sup>	89	6.31	<0.12	NA
	NE25865	6.6	0.08	77	1.79	<0.12	NA
	NE25866	6.1	0.28	77	11.1	<0.12	NA
	Lab Composite	NA	59.8 mg/kg <sup>3</sup>	NA	NA	NA	NA
S1B	NE25872	13.21	14.9 mg/kg <sup>3</sup>	83	9.40	0.268	NA
	NE25873	12.09	0.15 mg/kg	80	12.0	<0.12	NA
	NE25874	12.92	0.08 mg/kg	82	10.6 <sup>3</sup>	<0.12 <sup>3</sup>	NA
	NE25875	12.32	0.18 mg/kg	81	12.7	<0.12	NA
	NE25876	11.52 <sup>1</sup>	0.06 mg/kg	80	13.5	<0.12	NA
	NE25877	11.6	<0.01	77 <sup>3</sup>	12.6	<0.12	NA
	NE25878	13.2	46.2	77	9.69	0.201	NA
	NE25879	13.1	0.91	78	9.58	<0.12	NA
	NE25880	12.9	<0.01	77	10.4	<0.12	NA
	NE25881	12.1	0.11	80	12.1	<0.12	NA
	NE25882	13.1	<0.01	80	8.81 <sup>3</sup>	<0.12 <sup>3</sup>	NA
	NE25883	13.25	0.11 mg/kg	85	9.22	<0.12	NA
S2A	NE25867	12.3	4150 mg/kg <sup>3</sup>	78	3.26	1.84	NA
	NE25868	12.0	3860 mg/kg <sup>3</sup>	89 <sup>3</sup>	2.05	0.691	NA
	NE25869	12.58	5470 mg/kg <sup>3</sup>	85 <sup>3</sup>	3.03	3.74	NA
	NE25870	11.77	952 mg/kg	87	1.78 <sup>3</sup>	2.76 <sup>3</sup>	NA
S2B	NE25890	5.3	<0.005	63	28.3	<0.12	NA
	NE25892	NR	0.08	31 <sup>3</sup>	71.9 <sup>3</sup>	0.215 <sup>3</sup>	NA
S3A	NE25893	13.42	0.11 mg/kg	95	2.06	<0.12	NA
	NE25894	13.44	0.08 mg/kg	94	2.11	<0.12	NA
	NE25895	13.2	0.09	89	2.53	<0.12	NA
	NE25896	13.2	<0.01	84	2.66	<0.12	NA
S3B	NE25790	6.2	0.11	88	1.85	<0.12	NA
	NE25791	6.2	0.07	92	6.03	<0.12	NA
S4A	NE25897	13.2	0.10	84	8.99 <sup>3</sup>	<0.12 <sup>3</sup>	NA
	NE25898	13.2	<0.01	80 <sup>3</sup>	8.61	<0.12	NA
	NE25899	13.18	0.06 mg/kg	87	8.06	<0.12	NA
	NE25900	13.15	0.05 mg/kg	86	8.08	<0.12	NA

**Table 8. ANALYTICAL RESULTS**  
**CES Environmental Services**  
**Houston and Port Arthur, Texas**

NEIC Station No.	NEIC Tag No.	pH <sup>1</sup>	Sulfide <sup>2</sup> (mg/L except as noted)	Water (percent by weight)	Carbon (percent by weight)	Sulfur (percent by weight)	Anions (percent by weight)
S4B	NE25796	NR	0.18	<3	84.7 <sup>3</sup>	0.558 <sup>3</sup>	NA
	NE25797	NR	0.55	<3	82.5 <sup>3</sup>	0.581 <sup>3</sup>	NA
S5A	NE25884	13.55	0.10 mg/kg	91	4.50	<0.12	NA
	NE25885	13.54	0.11 mg/kg	96	4.46	<0.12	NA
	NE25886	13.3	<0.01	81 <sup>3</sup>	4.93	<0.12	NA
	NE25887	13.3	0.09	84	4.97	<0.12	NA
S6A	NE25902	6.2	<0.005	20	29.6	<0.12	NA
	NE25907	5.7	<0.005	17 <sup>3</sup>	29.5	<0.12	NA
S7A	NE25908	5.5	<0.005	23	26.2	<0.12	NA
	NE25910	5.5	<0.005	27	27.0	<0.12	NA
S8A	NE25788	7.4	<0.005	12	31.5 <sup>3</sup>	<0.12 <sup>3</sup>	NA
	NE25901	7.0	<0.005	12	31.9	<0.12	NA
S9A	NE25798	4.3	0.37	90	4.88	<0.12	NA
	NE25799	3.4	<0.005	87	1.54	<0.12	NA
S10A	NE25803	5.3	4.10 mg/kg	67	18.2	0.214	NA
	NE25805	5.3	7.13	73	14.4	0.188	NA
	NE25806	5.6	20.8 mg/kg	87	11.0	0.153	NA
	NE25811	5.4	9.07	73	14.8	0.149	NA
	NE25812	5.	2.51	66	18.3	0.210	NA
	NE25814	5.6	13.3 mg/kg	83	9.67	0.150	NA
	Lab Composite	NA	4.51 mg/kg <sup>3</sup>	NA	NA	NA	NA
S1C-01	NE25821	<1.0	<0.004	55	0.59	<0.12	25.1 chloride
	NE25822	<1.0	<0.004	55	<0.26	<0.12	26.0 chloride
S1C-02	NE25819	<1.0	<0.004	58	0.27	<0.12	24.8 chloride
	NE25820	<1.0	<0.004	56	0.28	<0.12	25.3 chloride
S1C-03	NE25845	<1.0	<0.004	62	<0.26	<0.12	16.2 chloride
	NE25846	<1.0	<0.004	66	<0.26	<0.12	16.5 chloride
S1C-04	NE25847	<1.0	<0.004	62	<0.26	<0.12	16.3 chloride
	NE25848	<1.0	<0.004	64	<0.26	<0.12	16.7 chloride
S1C-05	NE25853	<1.0	<0.004	59	0.30	<0.12	16.2 chloride
	NE25854	<1.0	<0.004	66	0.30	<0.12	16.3 chloride
S1C-06	NE25857	<1.0	<0.004	65	<0.26	<0.12	31.3 chloride
	NE25858	<1.0	<0.004	65	<0.26	<0.12	31.6 chloride

**Table 8. ANALYTICAL RESULTS**  
**CES Environmental Services**  
**Houston and Port Arthur, Texas**

NEIC Station No.	NEIC Tag No.	pH <sup>1</sup>	Sulfide <sup>2</sup> (mg/L except as noted)	Water (percent by weight)	Carbon (percent by weight)	Sulfur (percent by weight)	Anions (percent by weight)
S1C-07	NE25859	<1.0	<0.004	53	0.36 <sup>3</sup>	4.44 <sup>3</sup>	15.3 chloride <sup>3</sup> 12.7 sulfate <sup>3</sup>
	NE25860	<1.0	<0.004	50	0.35	4.39	15.7 chloride <sup>3</sup> 13.1 sulfate <sup>3</sup>
S1C-08	NE25963	<1.0	<0.004	54	<0.26	<0.12	25.6 chloride
	NE25964	<1.0	<0.004	56	0.59	<0.12	26.4 chloride
SS1	NE25891	NA	NA	64	NA	NA	NA

mg/L = milligrams per liter, mg/kg = milligrams per kilogram, NR = Not reported, NA = Not Analyzed

<sup>1</sup> All pH values except NE25876 are the average of multiple measurements. There was only enough sample remaining from other tests for one pH measurement on NE25876. A solid waste that is an aqueous liquids with pH values less than or equal to 2.0, or greater than or equal to 12.5 exhibits the RCRA hazardous waste characteristic of corrosivity (EPA Hazardous Waste Number D002).

<sup>2</sup> The uncertainty of the sulfide determinations for station S2A is 6 percent, expressed as a relative standard deviation. The uncertainty is 22 percent for the other sulfide determinations.

<sup>3</sup> Analytical result is the mean of triplicate measurements.

## WATER CONTENT DETERMINATION

Water content was measured to assist with the determination of hazardous waste characteristics of the samples. The phase 1 samples were diluted in methanol, and then introduced into the (b) (6), (b) (7)(C) titration apparatus by vaporization. For the phase 2 samples, the methanol dilutions were introduced directly into the (b) (6), (b) (7)(C) titration apparatus. Using sample introduction by vaporization for the phase 1 samples minimized interferences from hydroxide in the samples. This procedure was not necessary for the phase 2 samples. The results of the water content determinations are summarized in Table 8.

## DATA QUALITY SUMMARY FOR WATER CONTENT DETERMINATION

Quality control measures for the water content determinations included methanol blanks, replicate measurements, analysis of pure water, analysis of standards with low water content, and known additions. In order to evaluate possible interference from hydroxide, solutions of potassium hydroxide were prepared and analyzed. The results showed that the vaporization technique eliminated hydroxide interference. Calibration verification and continuing calibration verification were done with standards prepared from pure water.

The average standard deviation for replicate determinations of percent water was 1.9 weight percent with a range of standard deviations of 0.12 to 7.3 percent water. The range of relative standard deviations was from 0.66 to 9.1 percent, with an average relative standard deviation of 3.0 percent.

## SULFIDE DETERMINATION

Sulfide was determined using Conway diffusion cells that allow determination of the amount of sulfide that diffuses through the gas phase from the sample into a trapping solution. The sample is placed in the central well of the circular cell, and the trapping solution is placed in the outer chamber. The sample is adjusted to a pH between 2 and 3 before capping the cell and allowing the sulfide to diffuse for 3½ hours. The trapping solution is analyzed for sulfide using a flow injection analyzer with a gas diffusion membrane and amperometric detection. The results of the sulfide determinations are summarized in Table 8. Table 8 includes results for a composite of the station S1A samples and the S10A samples as well as individual results for all of the samples received.

## DATA QUALITY SUMMARY FOR SULFIDE DETERMINATION

Quality control measures for the sulfide determinations included blanks, replicate determinations, and known additions or spikes. Barium sulfide from two sources was used to



prepare standards, and for independent calibration verification. Standards were carried through the diffusion process as if they were samples. Sample measurements were confirmed with a sulfide ion selective electrode. Continuing calibration verification was performed for the flow injection analyzer.

The estimated uncertainty in the sulfide determinations is based on the replicate measurements. The estimated uncertainty for samples that range from three times the detection limit to 200 mg/kg or mg/L, expressed as a relative standard deviation, is 22 percent. For measurements over 900 mg/kg, the estimated uncertainty, expressed as a relative standard deviation, is 6 percent.

## **CARBON AND SULFUR DETERMINATIONS**

NEIC was requested to determine the sample concentration of total sulfur, which includes sulfide, hydrogen sulfide, organic sulfides, and inorganic sulfur forms such as sulfate. The instrument used for the sulfur determinations also determines total carbon at the same time. Carbon concentrations, along with water content determinations, can be used to understand the composition of a sample.

After being placed in a small ceramic combustion boat, samples are placed in a hot oven and combusted in a stream of oxygen. The carbon dioxide and the sulfur dioxide produced are measured by their absorption of infrared radiation. The results of the carbon and sulfur determinations are summarized in Table 8. The results are reported as percent carbon or sulfur by weight.

## **DATA QUALITY SUMMARY FOR CARBON AND SULFUR DETERMINATIONS**

Quality control measures for carbon and sulfur determinations include the analysis of replicates, blanks, and standard reference materials. Standards were used to calibrate the instrument, independently verify the calibration, and verify continuing calibration. The limit of detection for sulfur was determined from multiple blank determinations, and was calculated as 0.12 weight percent for phase 1 samples and 0.094 weight percent for phase 2 samples. The former, more conservative, value was used for samples in which sulfur was not detected, as reported in Table 8. The uncertainty was estimated from the quality control measures. For carbon, the uncertainty, expressed as a relative standard deviation, is equal to 7 percent. For sulfur, also expressed as a relative standard deviation, the uncertainty is 8 percent.

## ANALYSIS OF AIR CANISTER SAMPLES

Air samples were collected in stainless steel canisters. One project sample, NEIC tag No. NE25871 from station S2A, and one background sample, NEIC tag No. NE25912, were received and analyzed. They were analyzed by gas chromatography/mass spectrometry using an auto sampler designed specifically for canister samples. The first goal was to determine if hydrogen sulfide was present. No hydrogen sulfide was detected above the method detection limit in the project sample or in the background sample. The results are summarized in **Table 9**.

**Table 9. ANALYTICAL RESULTS FOR AIR CANISTER SAMPLES**  
**CES Environmental Services**  
**Houston & Port Arthur, Texas**

NEIC Tag No.	Sample Description Date Collected	Compound	Concentration (ppmv)
NE25871	Station S2A 8/5/2009	Hydrogen sulfide	< 0.13
		Dimethyl disulfide	64.6 <sup>1</sup>
		Carbonyl sulfide	< 11
		Dimethyl sulfide	< 11
		Methyl mercaptan	< 11
NE25912	Background 8/6/2009	Hydrogen sulfide	< 0.002
ppmv = parts per million by volume <sup>1</sup> Analytical result is the mean of triplicate measurements.			

The total ion chromatogram in the hydrogen sulfide analysis revealed the presence of many other compounds. The second goal was to identify and quantify some of these compounds. Some were tentatively identified, but their presence could not be confirmed because reference standards were not available. A reference standard containing several sulfur-containing compounds was purchased and used to determine if the compounds in the standard were present in the sample, and, if so, at what concentrations. Using this reference standard, it was determined that dimethyl disulfide was present in the sample, as shown in Table 9. Three other compounds in the standard that are listed in Table 9 were not in the sample. The quantitation limits are shown in the table.

## DATA QUALITY SUMMARY FOR AIR CANISTER ANALYSES

Quality control measures for the air canister analyses included initial calibration of the instrument response, internal standards, surrogate standards, independent calibration verification, continuing calibration verification, laboratory blanks, and additional verification of instrument

performance. No hydrogen sulfide was detected in the laboratory blanks. The field background sample was exhausted during the initial hydrogen sulfide determinations, so it could not be compared to the purchased standard. However, examination of the data from the initial analyses of the field background sample did not show the presence of any of the sulfides or the mercaptan in the purchased standards.

The relative standard deviation of replicate injections of the sample for dimethyl disulfide was 1.7 percent. Control sample and spike sample results indicate the reported value of 64.6 parts per million by volume (ppmv) underestimates the actual value by 30 to 45 percent.